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(54) Title: THERMOPLASTIC SUPERABSORBENT POLYMER BLEND COMPOSITIONS AND THEIR PREPARATION

(57) Abstract: An extrudable thermoplastic superabsorbent polymer blend composition is disclosed. The blend compositions are especially well suited for preparation of extruded or molded articles such as monolayer film, multilayer film, nonwoven web, sheet, foams, prother, multilayer laminate, fibers, tubes, rods or pipes which are well suited to preparation of power and communication cables or disposable absorbent articles such as diapers, sanitary napkins, tampons, incontinence products, hospital gowns or bed pads.

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## THERMOPLASTIC SUPERABSORBENT POLYMER BLEND COMPOSITIONS AND THEIR PREPARATION

The present invention relates to a thermoplastic polymer blend composition  
5 comprising a superabsorbent polymer and method of preparation thereof.

Superabsorbent polymers are well-known materials that are used in a variety of applications ranging from personal care articles such as diapers to water barrier applications in the construction industry to water blocking agents in communications cables to liquid  
10 absorbers in food packaging systems. These polymers are known to absorb several times their weight of, for example, moisture, water, saline solution, urine, blood, and serous body fluids.

One of the challenges of using superabsorbent polymer particles within an absorbent device is the containment or fixation of the superabsorbent polymer particles. Depending on the particular absorbent device, different approaches to contain or fix the superabsorbent  
15 polymer particles have been taken. For example, disposable absorbent products such as diapers, sanitary napkins, tampons, and incontinence products, typically comprise a matt or batt wrapped with a liner wherein the batt usually comprises the superabsorbent polymer in particulate form, see U.S. Patent No. 3,670,731. However, loss of particles and/or redistribution of the particles within the device, sometimes referred to as shakeout, often  
20 occurs.

An attempt to reduce shakeout is taught in U.S. Patent No. 4,806,598 which discloses nonwoven webs made from a thermoplastic polymer composition comprising a polyoxyethylene superabsorbent comprising a soft segment bonded to a hard segment through a reaction with a third segment and a thermoplastic polymer. However, there is  
25 little interaction between the polyoxyethylene superabsorbent and the thermoplastic polymer and the blends are not stable with regard to phase separation. Further, webs made from the thermoplastic polymer composition do not demonstrate adequate wet strength and attempts to improve the wet strength of the webs by replacing some of the thermoplastic polymer composition with a low density polyethylene results in substantially decreasing the  
30 water absorbency of the web.

In power and communication cable applications different approaches have been tried to bind or fix superabsorbent polymers as water-blocking agents. For example, see U.S. Patent No. 4,966,809 which discloses water-blocking tapes made by mixing a

superabsorbent polymer and a polymeric binder and then spreading the mixture on nonwoven fabrics, see U.S. Patent No. 5,461,195 which discloses a superabsorbent polymer mixed with a thixotropic agent to form a gel which is used to fill the spaces between the wires of the cable or see U.S. Patent No. 5,925,461 which discloses strengthening members or buffer tubes coated or impregnated with a hot melt adhesive comprising a superabsorbent.

Mixtures of superabsorbent polymers and binders are characterized by a number of disadvantages and/or limitations, such as manufacturing and operating temperature limitations, lack of adhesion to the substrates to which the mixture is applied, and delaminating when the article is pulled in the tensile direction, that contribute to abrasion when the article is being fabricated. Further, tapes add additional components in the construction of cables causing considerable unwanted increases in their costs and diameters. Cables using filler gels are characterized by a number of disadvantages and/or limitations such as manufacturing and operating temperature limitations, formation of voids which lead to paths of water migration, and difficulties meeting industry standards.

Other methods to bind superabsorbent polymers are known. For example see, U.S. Patent No. 5,516,585 which discloses a method of coating discontinuous fibers with a thermoset binder material which binds particles of superabsorbent wherein the discontinuous fibers are formed into a web. In a method described in U.S. Patent No. 4,392,908 superabsorbent polymer particles are coated with a thermoplastic resin and fixed to a water-absorbent substrate by applying heat to soften the thermoplastic coating of the particles and pressing the particles and substrate to cause the particles to bind to the substrate. These methods are expensive requiring specialized equipment and/or many steps and have limited commercial applicability.

Further, films and laminates of superabsorbent polymers have been made from solutions of superabsorbent polymers followed by heating and/or removing the solvent. For examples of cross-linked superabsorbent polymer films and laminates see U.S. Patent Nos. 3,926,891, 4,076,673 and 4,117,184. For examples of non-cross-linked superabsorbent polymer films see U.S. Patent Nos. 3,935,099, 3,997,484 and 4,090,013. U.S. Patent No. 3,669,103 describes a method to make thin foamed polyurethane thermoset sheet comprising superabsorbent polymer particles. Unfortunately, these methods of forming films, laminates and sheet are impractical for large-scale commercial use.

It would be desirable to have a superabsorbent polymer composition with improved containment of superabsorbent polymer particles for use in absorbent devices such as personal-care articles and cable wrap components while maintaining good absorptive properties. Further, it would be desirable for such a superabsorbent polymer composition to be easily and conveniently shaped into a variety of useful forms, especially on a commercial scale.

The present invention is such a composition. It is a thermoplastic superabsorbent polymer blend composition comprising (a) a superabsorbent polymer (b) a thermoplastic resin and optionally (c) a surfactant wherein components (a) and (b) interact with each other ionically or covalently and the blend composition can be formed by extrusion, for example, into film, sheet, laminates, foams, profiles and injection molded articles.

In another aspect, the present invention is a process for preparing the abovementioned extrudable thermoplastic superabsorbent polymer blend composition.

In a further aspect, the present invention involves a method of extruding or molding the abovementioned extrudable thermoplastic superabsorbent polymer blend composition.

In yet a further aspect, the invention involves extruded (for example, film, sheet, foam, and laminates) or molded articles of the abovementioned extrudable thermoplastic superabsorbent polymer blend composition.

In yet a further aspect, the invention involves articles comprising extruded or molded articles of the abovementioned extrudable thermoplastic superabsorbent polymer blend composition.

The blend compositions and extruded and molded articles of the present invention may be employed in a wide variety of uses as are known in the art, such as, for example, the assembly or construction of cable wrap components and various disposable absorbent articles, such as sanitary napkins, disposable diapers, hospital gowns, and bed pads.

The superabsorbent water-swellaable or lightly cross-linked hydrophilic polymers suitably employable in the present invention can be any of the known hydrophilic polymers that are capable of absorbing large quantities of fluids. These polymers are well known in the art and are widely commercially available.

Examples of some suitable polymers and processes, including gel polymerization processes, for preparing superabsorbent polymers are disclosed in U.S. Patent Nos. 3,997,484; 3,926,891; 3,935,099; 4,090,013; 4,093,776; 4,340,706; 4,446,261; 4,683,274; 4,459,396; 4,708,997; 4,076,663; 4,190,562; 4,286,082; 4,857,610; 4,985,518; and

5,145,906. In addition, see Buchholz, F.L. and Graham, A.T., "Modern Superabsorbent Polymer Technology," John Wiley & Sons (1998) and Lisa Brannon-Peppas and Ronald S. Harland, "Absorbent Polymer Technology" Elsevier (1990).

Preferred superabsorbent polymers are prepared from water-soluble  
5  $\alpha,\beta$ -ethylenically unsaturated monomers such as monocarboxylic acids, vinyl polycarboxylic acids, acrylamide and their derivatives. More preferred superabsorbent polymers are cellulosic or starch-graft copolymers, such as starch-g-poly(acrylonitrile), and starch-g-poly(acrylic acid); polyacrylamides; polyvinyl alcohols; poly(acrylic acids); high  
10 molecular weight polymers, preferably cross-linked, of ethyleneoxide (EO) and propyleneoxide (PO); copolymers of sulfonic acid group containing monomers, such as vinyl sulfonic acid, sodium sulfoethyl methacrylate, 2-Acrylamido-2-Methylpropane-sulfonic acid or the sodium salt (AMPS).

Most preferred superabsorbent polymers are crosslinked, partially neutralized and/or surface treated. Preferably, the level of crosslinking is selected to give the desired swelling  
15 characteristics for the particular application. Generally, the degree of neutralization is from 30 to 100 percent, more preferably from 50 to 80 percent. Neutralization with a basic substance containing a Group I metal ion, such as sodium, is preferred. A preferred surface treatment consists of a post polymerization reaction to effect the surface crosslinking of the superabsorbent polymer.

20 The amount of the superabsorbent polymer to be included in the thermoplastic superabsorbent polymer blend composition according to the present invention will vary depending, for example, upon the type of superabsorbent polymer used, the type of thermoplastic resin used, the desired extruded or molded product, the extruded or molded product's end use application, the desired level of blocking, absorbing or stopping the  
25 migration of water and/or other fluids in the end use application.

The superabsorbent polymer is present in an amount equal to or greater than about 1 part per weight, preferably equal to or greater than 5 parts per weight, more preferably equal to or greater than 10 parts per weight, even more preferably equal to or greater than 15 parts per weight and most preferably equal to or greater than 20 parts per weight based on the  
30 weight of the thermoplastic superabsorbent polymer blend composition. The amount of superabsorbent polymer is present in an amount equal to or less than 70 part per weight, preferably equal to or less than 65 parts per weight, more preferably equal to or less than 60 parts per weight, even more preferably equal to or less than 55 parts per weight and most

preferably equal to or less than 50 parts per weight based on the weight of the thermoplastic superabsorbent polymer blend composition.

In addition to a superabsorbent polymer, the blend composition of the present invention contains at least one thermoplastic resin that interacts (that is, ionically,  
5 covalently) with the superabsorbent polymer. For example, a thermoplastic resin having an acyl groups which can undergo nucleophilic attack resulting in a substitution reaction in which a leaving group, such as -OH, -Cl, -OOCR, -NH<sub>2</sub> or -OR, is replaced by another basic group present in the superabsorbent polymer. Another example is a thermoplastic resin containing carbonyl groups that can undergo a nucleophilic attack gaining a proton  
10 and adding another basic group present in the superabsorbent polymer. Under these conditions the reaction product of the thermoplastic resin and superabsorbent polymer may form a uniform and/or co-continuous non-separating polymer blend.

Preferred thermoplastic resins have functional groups such as acyl or carbonyl groups (for example,  $\alpha,\beta$ -unsaturated carbonyl compounds, hydroxy acids, dicarboxylic  
15 acids, keto acids, anhydrides, carboxylic acids, aldehydes, ketones, acid halides, esters, amides, etc.), sulfonyls, sulfonyls halides, ethers, phenols, aryl halides, epoxides, carbohydrates, alcohols, azides, and amines.

The preferred thermoplastic resins are acrylic polymers, with polyacrylic acid (PAA), ethylene and acrylic acid copolymers (EAA), ethylene, t-butylacrylate and acrylic  
20 acid terpolymer (EtBAAA), ethylene and methacrylic acid copolymers (EMAA), ionomers of ethylene and methacrylic acid copolymers especially the sodium and zinc ionomers, ethylene, vinyl acetate and carbon monoxide terpolymers (EVACO), ethylene and carbon monoxide copolymers (ECO), ethylene, acrylic acid and carbon monoxide terpolymers (EAACO), ethylene, n-butylacrylate and carbon monoxide terpolymers (EnBACO) and  
25 blends thereof being most preferred.

The most preferred thermoplastic resins are 1) an EAA copolymer, wherein the EAA copolymer may be a blend of two or more EAA copolymers, preferably having a composition from 10 to 20 weight percent acrylic acid based on the weight of the copolymer and a melt flow rate (MFR) from 100 to 200 grams per 10 minutes (g/10 min.)  
30 under conditions of 190°C and an applied load of 2.16 kg., 2) ionomers of EMAA, preferably the zinc ionomer, 3) EVACO, preferably having a carbon monoxide content of at least 9 percent based on the weight of the terpolymer or 4) blends thereof.

The thermoplastic resin is present in an amount equal to or greater than 30 part per weight, preferably equal to or greater than 35 parts per weight, more preferably equal to or greater than 40 parts per weight, even more preferably equal to or greater than 45 parts per weight and most preferably equal to or greater than 50 parts per weight based on the weight of the thermoplastic superabsorbent polymer blend composition. The amount of thermoplastic resin is present in an amount equal to or less than 99 parts per weight, preferably equal to or less than 95 parts per weight, more preferably equal to or less than 90 parts per weight, even more preferably equal to or less than 85 parts per weight and most preferably equal to or less than 80 parts per weight based on the weight of the thermoplastic superabsorbent polymer blend composition.

It should be apparent to those having ordinary skill in the art that the present invention contemplates blends containing two or more superabsorbent polymers and/or blends of two or more thermoplastic resins (for example, EAA/EVACO, EMAA/EAA, a first EAA/a second EAA).

While the blend compositions of the present invention contain at least one superabsorbent polymer, such blend compositions may or may not be superabsorbent, depending upon the level and absorbency of the superabsorbent polymer in the blend composition and the availability of the superabsorbent polymer to aqueous media.

The blend compositions of the present invention can be further blended with other thermoplastic polymers, preferably low density polyethylene (LDPE), linear low density polyethylene (LLDPE), very low density polyethylene (VLDPE), polypropylene (PP), polystyrene (PS), ethylene and methylacrylate copolymer (EMA), ethylene and ethylacrylate copolymer (EEA), ethylene and n-butylacrylate copolymer (EnBA), polyethylene grafted with maleic anhydride grafted (PE g-MAH), ethylene and vinyl acetate copolymer (EVA), ethylene and vinyl acetate copolymer grafted with maleic anhydride grafted (EVA g-MAH), or combinations thereof.

The blend compositions of the present invention may further comprise additional additives commonly used in compositions of this type such as lubricants, extenders, compatibilizers, plasticizers, low and high molecular weight waxes, surfactants, stabilizers, pigments, carbon black and fillers such as talc, titanium dioxide (TiO<sub>2</sub>), calcium carbonate (CaCO<sub>3</sub>), magnesium oxide (MgO), and mica.

The blend compositions of the present invention may further be blended with a solvent to form a dispersion or paste. One skilled in the art can readily choose the type and amount of solvent depending on the particular end use.

As used herein, the phrase "extrudable thermoplastic superabsorbent polymer blend composition" means that: (1) the blend composition is melt processable in an extrusion, injection molding and/or blow molding process, (2) the extrudate is either commuted to pellets or directly extruded or molded by extrusion fabrication technique, (3) the pellets have a measurable melt flow rate, melt draw rate and melt strength sometimes referred to as melt tension and (4) the pellets can be re-extruded by an extrusion fabrication techniques. Preferably, the blend compositions of the present invention do not cause plugging, die-face build up, surging, melt fracture, pinholes, tearing and/or poor extrudate properties (that is, strand dropping, delamination) during the extrusion process.

A melt indexer is used to determine melt flow rate (MFR), melt tension and draw down rate. MFR is determined by ASTM D 1238; the run conditions (that is, temperature and applied load) depend upon the thermoplastic resin used. Melt tension is determined from a load cell attached at the bottom of the melt indexer which measures the load required to pull the extrudate from the die of the melt indexer to a take up reel at some given speed measured in feet per minute (fpm). The draw down rate (fpm) is determined by how fast the extrudate coming out of the melt indexer can be pulled before it breaks. When MFR conditions are selected to give a MFR between 0.1 and 300 g/10 min. the thermoplastic superabsorbent polymer blend composition has a melt draw down rate between 5 and 100 fpm and a melt tension between 0.1 and 10.

The components of the extruded blend composition can be co-continuous or separate phases (one being continuous and one or more being dispersed therein) as long as phase separation does not have a significant deleterious effect on the melt processability or performance of the blend composition.

Preferred extrusion fabrication techniques include preparing melt blown or cast films; extrusion coating; (co)extruding nonwoven webs, including spun bond nonwoven webs, melt blown nonwoven webs, or composites comprising combinations thereof, sheets, foams, profiles, multilayer laminates, fibers including monofilament fibers and bicomponent monofilament fibers, tubes, rods or pipes; blow molding articles; injection molding articles (including solid, co-injection, structural foam and gas assist injection molding). Preferred nonwoven webs comprise spun bond nonwoven webs comprising one



or more bicomponent fiber, melt blown nonwoven webs comprising one or more bicomponent fiber, and a composite structure comprising at least one layer of one or more spun bond nonwoven web and at least one layer of one or more melt blown nonwoven web wherein one or more layers of the composite comprise bicomponent fibers.

5       The thermoplastic superabsorbent polymer blend compositions of the present invention can be extruded into foam using a chemical or physical blowing agent. Further, the thermoplastic superabsorbent polymer can be blended with other miscible or compatible thermoplastic polymers such as LDPE, LLDPE, VLDPE, PP, PS, EEA, EMA, ENBA, PE g-MAH, EVA, or EVA g-MAH. One skilled in the art can choose the type and amount of  
10       blowing agent as well as other polymers to blend with the thermoplastic superabsorbent polymer for the particular end use in order to modify the cell size, structure, porosity, microcellular nature and absorbency characteristics of the thermoplastic superabsorbent polymer foam as desired.

      The blend compositions to make foam may further comprise additional additives  
15       commonly used in compositions of this type such as lubricants, extenders, nucleators, compatibilizers, plasticizers, low and high molecular weight waxes, surfactants, stabilizers, pigments, carbon black and fillers such as talc,  $\text{TiO}_2$ ,  $\text{CaCO}_3$ ,  $\text{MgO}$ , and mica.

      Further, extruded pellets or sheet can be compression molded; calendered; vacuum formed or thermoformed. Preparation of the thermoplastic superabsorbent polymer blend  
20       compositions of this invention can be accomplished by any suitable mixing means known in the art. Typically the components and any additional additives are blended in a tumbler or shaker in powder, particulate and/or pellet form with sufficient agitation to obtain thorough distribution thereof. The dry-blended formulation can further be subjected to shearing stresses at a temperature sufficient to heat soften and melt-mix the polymers, for example in  
25       an extruder, with or without a vacuum, or other mixing apparatuses (for example, a Banbury mixer, roller mill, Henschel mixer, a ribbon blender). Further, additional powder, particulate and/or liquid additives may be added to the composition during the mixing process. Such melt-mixed material can be extruded to make the finished article (that is, film, sheet, foam, profile) or recovered in the form of a pellet, powder or flake, preferably a  
30       pellet. The extrudate may be commuted to pellets by any conventional means such as a strand chopper or an underwater die face cutter.

      The extrudate from the melt-mixing may be cooled by any method known in the art, such as air cooled, gas cooled, belt cooled, and liquid cooled by passing through a liquid

bath. Preferably a stainless steel belt cooler, for example manufactured by Sandvik Process Systems, Sweden or a Compact Conti Cooler manufactured by BBA AG, Switzerland, or an aqueous liquid bath, preferably where the pH is less than 1.0 or an aqueous liquid bath with a water hardness of greater than 25 French Degrees, more preferably an aqueous liquid bath with a specific gravity greater than 1.05 as measured with a desitometer is used. The aqueous bath preferably contains a saturated salt solution containing a Group 1 metal ion, preferably sodium, such as sodium chloride (NaCl), sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>), and sodium bicarbonate (NaHCO<sub>3</sub>).

Further, to minimize the effect of the water on the superabsorbent compound it has been discovered that maintaining the temperature of the liquid bath less than 23°C and preferably less than 20°C effectively cools the pellets without grossly activating the superabsorbent polymer in the blend composition.

It has further been found that when using an underwater pelletizer, optimizing the transfer pipe length from the underwater pelletizer to the separating dryer minimizes the activation of the superabsorbent polymer in the blend composition.

Blowing cool air in the pellet-collecting vessel, such as the use of a fluidized bed cooler, to drive away the remaining moisture on the pellets further improves the drying process.

It has been found using a process comprising an underwater die face cutter, a saturated NaHCO<sub>3</sub> solution having a specific gravity greater than 1.05 and a temperature less than 20°C and blowing cool air in the pellet-collecting vessel yields an extrudable thermoplastic superabsorbent polymer blend composition in a free flowing plastic pellet form having a moisture content ranging from 0.2 to 4 weight percent depending on the superabsorbent polymer, concentration of superabsorbent polymer in the blend composition and the base thermoplastic resin, wherein moisture weight percent is based on the weight of the blend composition.

The melt-mixed material (powder, flake or pellet) can be re-extruded or molded to make the finished article. Dry blends of the blend compositions can also be directly injection molded or metered into another melt fabrication process without pre-melt-mixing.

The extrudable thermoplastic superabsorbent polymer blend compositions of the present invention are useful in the pellet, flake or powder form for use in cat litter, solidified gases/fluids, gelled ice, soil conditioner, frost control, agricultural delivery systems, gelled biohazards, spill control, for the fabrication of articles such as foams, such as closed, semi-

porous or microcellular or open cell, bicomponent fibers and waterproof or waterblocking coating systems, thick film or sheet for such applications as disposable absorbent articles, such as sanitary napkins, disposable diapers, hospital gowns, and bed pads, films for such applications as moisture sensitive systems, moisture, such as water, absorbing structures, for example in packaging, transportation, and construction applications, diaper backing, meat trays, carpet backing or power and communication cable water-blocking tapes, film for laminate structures such as laminated foam structures, laminated non-woven structures, film for laminates for such applications as cable shielding tapes for use in power cables or communication cables, such as fiber optical cables, copper pair cables, and coaxial cables as disclosed in U.S. Patent Nos. 3,795,540, 4,449,014, 4,731,504 and 4,322,574.

It is further desirable that when the present invention is used in the construction of cables for example, power cables and communication cables, such as fiber optical cables, copper pair cables, coaxial cables, the cables meet certain requirements of water penetration. Most desirably, a cable structure comprising an extrudable thermoplastic superabsorbent polymer blend composition resists penetration, sometimes referred to as water blocking, of water through the cable in the longitudinal direction.

To illustrate the practice of this invention, examples are set forth below.

#### Examples

##### Thermoplastic Superabsorbent Polymer Blend Compositions

In Comparative Examples A to ZZ and Examples 1 to 13 different thermoplastic resins are melt blended in a Brabender Plasticoder with CABLOC™ 850-13 a sodium polyacrylate superabsorbent polymer that is surface cross-linked having a particle size distribution of 1 to 300 micrometers available as a powder from Stockhausen and supplied by the Stewart Superabsorbents LLC. Unless otherwise noted, the ratio of superabsorbent polymer to thermoplastic resin is 40:60. The Brabender Plasticoder conditions are: Barrel temperature ranging from 275 to 420°F depending on the thermoplastic resin being used; Mixing revolutions per minute (RPM) are 80; and Mixing times range from 1.5 to 2 minutes. A melt indexer is used to determine the melt flow rate, melt tension and melt draw down rate of the polymer blends

Table 1 lists the compositions for Comparative Examples A to ZZ and Examples 1 to 13 and their properties. In Table 1 blend compositions which can be melt blended in some fashion and pressed into a shape or molded into a sheet or an article, but do not meet the criteria set forth herein as extrudable are designated not extrudable.

Comparative Examples AB to AN and Examples 14 to 17 are compounded on a WP ZK30 twin screw extruder. The SAP and the polymer are fed separately into the feed section of the extruder, the vent port of the extruder is open to the atmosphere and the extrudate is air-cooled.

- 5        The compositions of Comparative Examples AB to AN and Examples 14 to 17 and extruder temperatures are given in Table 2, the superabsorbent polymer is present in parts by weight based on the weight of the thermoplastic superabsorbent polymer blend composition. In Table 2 compositions that demonstrate die face build-up and/or plugging are designated "not" extrudable.

Table 1

Example	Com. Ex.	Thermoplastic Resin			Thermoplastic Superabsorbent Polymer Blend Composition					
		Grade	Supplier	Type	MFR, Condition	MFR, g/10 min	Melt Tension, units	Draw Rate, fpm	Extrudable	
	A	ALATHON™ M6060	Equistar	HDPE	E	5.20			No	
	B	LDPE 4005	Dow Chemical Co.	LDPE	E	1.98			No	
	C	LDPE 4012	Dow Chemical Co.	LDPE	E	4.5	0.8	<5	No	
	D	LDPE 681	Dow Chemical Co.	LDPE	E	0.72			No	
	E	DOWLEX™ 2247A	Dow Chemical Co.	LLDPE	E	1.33			No	
	F	ASPUN™ 6821	Dow Chemical Co.	LLDPE	B	11.2			No	
	G	ATTANE™ 4201	Dow Chemical Co.	VLDPE	E	0.417			No	
	H	ATTANE 4402	Dow Chemical Co.	VLDPE	E	1.16			No	
	I	AFINITY™ 1880	Dow Chemical Co.	INSITE™ PE	E	0.594			No	
	J	ENGAGE™ 8200	DuPont Dow	INSITE PE	E	3.26			No	
	K	PP 861	Montell	PP	L	7.6			No	
	L	PS 680	Dow Chemical Co.	PS	G	5.8			No	
	M	Chevron 2252-T	Chevron	EMA	E	0.42			No	
	N	Chevron 2255	Chevron	EMA	E	1.30			No	
	O	Chevron 1802	Chevron	EnBA	E	0.44			No	
	P	ENGAGE SM8400	Dow Chemical Co.	PE g-MAH/high MAH	E	0.28			No	
	Q	FUSABOND™ 190D	DuPont	EVA g-MAH/high MAH	E	0.5			No	
	R	FUSABOND 197D	DuPont	EVA g-MAH/high MAH	E	0.1			No	
	S	FUSABOND 226D	DuPont	LLDPE g-MAH/high MAH	E	0.1			No	
	T	FUSABOND 274D	DuPont	EPDM g-MAH/medium MAH	E	0.1			No	
	U	FUSABOND 413D	DuPont	PE g-MAH MAH	E				No	
	V	FUSABOND 423G	DuPont	EA terpolymer g-MAH/high MAH	E	1.43			No	
	W	FUSABOND 353D	DuPont	PP g-MAH/very high MAH	160 °C / 0.353 Kg	2.62			No	
	X	BYNEL™ E418	DuPont	Anhydride Modified EVA	E	2.41			No	

Table 1 Continued

Example		Com. Ex.	Thermoplastic Resin			Thermoplastic Superabsorbent Polymer Blend Composition				
			Grade	Supplier	Type	MFR, Condition	MFR, g/10 min	Melt Tension, units	Draw Rate, fpm	Extrudable
	Y		CXA 3101	DuPont	Acid/Acrylate Modified EVA	E	1.71			No
	Z		CXA 4105	DuPont	Anhydride Modified LLDPE	E	0.84			No
	AA		BYNEL 50E561	DuPont	Anhydride Modified PP	E	0.90			No
	BB		BYNEL 2174	DuPont	Anhydride Modified EA	E	0.75			No
	CC		PLEXAR™ 3	Equistar	Anhydride Modified EVA	E	1.24			No
	DD		PLEXAR 206	Equistar	Anhydride Modified HDPE	E	2.77			No
	EE		STEREON™ 841A	Firestone	SBS block copolymer	G	5.42			No
	FF		VECTOR™ 4211	Dexco Polymers	SIS block copolymer	G	11.58			No
	GG		VECTOR 4461	Dexco Polymers	SBS block copolymer	G	9.72			No
	HH		KRATON™ G1657	Shell	SEBS block copolymer	G	3.82			No
	II		KRATON FG1901X	Shell	SEBS block copolymer	G	0.25			No
	JJ		VECTOR 4411	Dexco Polymers	SIS block copolymer	G	18.2			No
	KK		Phillips DK-11	Phillips	SBS block copolymer	G	3.62			No
	LL		Phillips K-10	Phillips	SBS block copolymer	G	4.6			No
	MM		VECTOR 8508	Dexco Polymers	SBS block copolymer	G	3.1			No
	NN		ESI DE 200	Dow Chemical Co.	Ethylene-Styrene Interpolymer	G	4.75			No
	OO		ESI DS 201	Dow Chemical Co.	Ethylene-Styrene Interpolymer	G	5.6			No -
	PP		ELVAX™ 3180	DuPont	EVA, 28% VA	E	13.88			No
	QQ		ELVAX VOW	DuPont	EVA, 49% VA	B	1.5			No
	RR		GRILTEX™ 9	EMS Am. Grilon, Inc	CoPolyester hot melt adhesive	C	4.4			No
	SS		GRILTEX D 1519EGF	EMS Am. Grilon, Inc	CoPolyester hot melt adhesive	C	2.3			No
	TT		MACROMELT™ 6238	Henkel	Polyamide Resin	C				No
	UU		MACROMELT 6206	Henkel	Polyamide Resin	C	24.52			No
	VV		PHAE	Dow Chemical Co.	Thermoplastic Phenoxo Resin	E	7.5			No
	WW		LDPE 457	Dow Chemical Co.	ECO, 1% CO	E	0.33			No
1			ELVALOY™ HP441	DuPont	ENBACO	E	3.1	1.5	5	Yes
2			ELVALOY EP4924	DuPont	EVACO	E	7.28	0.5	20	Yes

Table 1 Continued

Example	Com. Ex.	Thermoplastic Resin			Thermoplastic Superabsorbent Polymer Blend Composition				
		Grade	Supplier	Type	MFR, Condition	MFR, g/10 min	Melt Tension, units	Draw Rate, fpm	Extrudable
3		A702	Chevron	EEA	E	2.8	1.0	5	Yes
	XX	PRIMACOR™ 3330	Dow Chemical Co.	EEA, 6.5% AA	E	2.2			No
4		PRIMACOR 1410	Dow Chemical Co.	EEA, 9.7% AA	E	0.72			Yes
5		PRIMACOR 1430	Dow Chemical Co.	EEA, 9.7% AA	E	2.43	1.0	5	Yes
6		PRIMACOR 3460	Dow Chemical Co.	EEA, 9.7% AA	E	8.98	0.6	20	Yes
7		XUS70751.17	Dow Chemical Co.	EEA, 20.5% AA	B	0.84	1.5	10	Yes
	YY	PRIMACOR 5980	Dow Chemical Co.	EEA, 20.5% AA	B	0.3			No
8		PRIMACOR blend (a)	Dow Chemical Co.	EEA, 15.1% AA	B	1.16	1.4	10	Yes
9		ESCOR™ ATX 325	Exxon	EMAAA	E	8.72	0.2	5	Yes
10		NUCREL™ 699	DuPont	EMAA	B	4.6	0.5	5	Yes
11		SURLYN™ 8660	DuPont	Na-EMAA ionomer	125 °C / 5.0 Kg	1.46	2.0	5	Yes
12		SURLYN 1702	DuPont	Zn-EMAA ionomer	E	6.0	0.9	45	Yes
13		SURLYN 1702 (b)	DuPont	Zn-EMAA ionomer	E	4.13	0.5	20	Yes
	ZZ	SURLYN 1702 (c)	DuPont	Zn-EMAA ionomer	E	2.58			No

(a) a 50/50 blend of PRIMACOR 3460 and PRIMACOR 5980

(b) a 50/50 blend of SURLYN 1702 and CABLOC 850-13

(c) a 40/60 blend of SURLYN 1702 and CABLOC 850-13

LDPE = low density polyethylene

LLDPE = linear low density polyethylene

VLDPE = very low density polyethylene

PP = polypropylene

PS = polystyrene

EMA = ethylene and methylacrylate copolymer

EnBA = ethylene and n-butylacrylate copolymer

PE = polyethylene

Q-MAH = grafted with maleic anhydride

EVA = ethylene and vinyl acetate copolymer

EPDM = ethylene propylene diene monomer

EA = ethylene and acrylate copolymer

SBS = styrene, butadiene and styrene block copolymer

SIS = styrene, isoprene and styrene block copolymer

SEBS = styrene, ethylene, butylene and styrene block copolymer

ESI = ethylene and styrene interpolymer block copolymer

ECO = ethylene and carbon monoxide copolymer

EnBACO = ethylene, n-butylacrylate and carbon monoxide terpolymer

EVACO = ethylene, vinyl acetate and carbon monoxide terpolymer

EEA = ethylene and ethyl acrylate copolymer

EAA = ethylene and acrylic acid copolymer

AA = acrylic acid

EMAA = ethylene and methacrylic acid copolymer

EMAAA = ethylene and methyl acrylate and acrylic acid copolymer

Na = sodium

Zn = zinc

Condition B = 125°C/2.16 kg

Condition C = 150°C/2.16 kg

Condition E = 190°C/2.16 kg

Condition G = 200°C/5.0 kg

Condition L = 230°C/2.16 kg

Table 2

Example	Com. Ex.	Thermoplastic Resin			Superabsorbent Polymer	Blend Composition, parts		Extruder Temperatures °F	Extrudable
		Grade	Supplier	Type		Resin	SAP		
	AB	LDPE 681	Dow Chemical Co.	LDPE	SAP-1	80	20	310-330	No
	AC	LDPE 681	Dow Chemical Co.	LDPE	SAP-1	75	25	310-330	No
	AD	LDPE 681	Dow Chemical Co.	LDPE	SAP-1	65	35	310-330	No
	AE	LDPE 681	Dow Chemical Co.	LDPE	SAP-1	60	40	310-330	No
	AF	ATTANE 4201	Dow Chemical Co.	VLDPE	SAP-2	80	20	335-370	No
	AG	ATTANE 4203	Dow Chemical Co.	VLDPE	SAP-1	73	27	321-350	No
	AH	ALATHON 6030 HPPE	Equistar	HDPE	SAP-2	80	20	335-370	No
	AI	DOWLEX 2045	Dow Chemical Co.	LLDPE	SAP-2	75	25	400-420	No
	AJ	ELVAX 3180	DuPont	EVA, 28% VA	SAP-1	60	40	250-260	No
	AK	Aqua Calk (a)	Sumitomo Seika Chemical Co., Ltd.	Polyethylene Oxide(a)	SAP-1	60	40	250-260	No
14	AL	PRIMACOR 3330	Dow Chemical Co.	EAA, 6.5% AA	SAP-1	60	40	310-330	No
		PRIMACOR 3460	Dow Chemical Co.	EAA, 9.7% AA	SAP-1	60	40	250-260	Yes
15	AM	PRIMACOR 5980	Dow Chemical Co.	EAA, 20.5% AA	SAP-1	60	40	250-260	No
		PRIMACOR blend (b)	Dow Chemical Co.	EAA, 15% AA	SAP-2	60	40	250-260	Yes
16	AN	PRIMACOR blend (b)	Dow Chemical Co.	EAA, 15% AA	SAP-2	50	50	250-260	No
		PRIMACOR 3460	Dow Chemical Co.	EAA, 6.5% AA	SAP-3	60	40	250-260	Yes
17		ELVALOY EP4924	DuPont	EVACO	SAP-4	60	40	250-260	Yes

(a) Aqua Calk is a thermoplastic, non-ionic, water-absorbent polymer, manufactured by cross-linking polyethylene oxide

(b) 50:50 blend of PRIMACOR 3460 and PRIMACOR 5980

SAP = superabsorbent polymer

SAP-1 is a polyacrylate based superabsorbent polymer available as CABLOC 1181 from Stockhausen having a particle size distribution from about 1 to about 50 micrometers

SAP-2 is a polyacrylate based superabsorbent polymer available as CABLOC 80HS from Stockhausen having a particle size distribution from about 1 to about 100 micrometers

SAP-3 is a polyacrylate based superabsorbent polymer available as DRYTECH 2035 from Dow Chemical Company having a particle size distribution from about 1 to about 500 micrometers

SAP-4 is a polyacrylate based superabsorbent polymer available as CABLOC 88HS from Stockhausen having a particle size distribution from about 1 to about 150



Comparative Examples AO to AW are different neat thermoplastic resins, Comparative Example AU is neat superabsorbent polymer CABLOC 850-13, AV is the neat superabsorbent polymer CABLOC 80HS, AW is the neat superabsorbent polymer CABLOC 88HS and Examples 18 to 30 are different thermoplastic resins compounded with a superabsorbent polymer. A ZSK 58 millimeter (mm) co-rotating bi-lobe twin screw extruder having a low shear mixing screw and 10 temperature zones is used. The superabsorbent polymer is fed using a side port powder screw feeder between zones 4 and 5. Mixing occurs in zone 6. The transition point between zone 8 and 9 is the vent port. There is a kneading mixing section prior to the vent port. The temperature range for the first 3 zones is from 65 to 120°F, for zones 4 and 5 it is from 240 to 255°F, for zones 6 to 8 it is from 320 to 335°F and for zones 9 and 10 it is from 270 to 330°F. The melt temperature is maintained at 310°F.

The blend compositions are extruded through a 24 hole underwater die having hole diameters of 0.110 inch into a liquid bath containing a  $\text{NaHCO}_3$  solution having a specific gravity of greater than 1.05 as measured with a desitometer with a temperature maintained below 20°C. A Gala underwater pelletizer with 3 cutting blades is used to pelletize the extrudate. The distance from the underwater pelletizer to the separating dryer is optimized to minimize the adsorption of water. Further, cool air is blown on the pellets in the pellet-collecting vessel driving away any remaining moisture on the pellets.

The absorption capacity in pure water (WAC) of the neat thermoplastic resins Comparative Examples AO to AT, the neat superabsorbent polymers Comparative Examples AU to AW and the thermoplastic superabsorbent polymer blend compositions Examples 18 to 30 is measured according to the following procedure: For the thermoplastic superabsorbent polymer blend compositions a sample determined to contain 1 gram of the superabsorbent polymer (based on the percent superabsorbent polymer in the blend composition) weighing  $W_1$  is placed in 1.5 liter of distilled water and is shaken on a shaker for 2 hours. The water is filtered from the swollen particles through a 75 micrometer sieve. The weight of the swollen particles ( $W_2$ ) is then measured. The amount of water absorbed,  $W_a$ , is  $W_2 - W_1$ . For the neat resins and neat superabsorbent polymers a sample weighing 1 gram is subjected to the same procedure described herein above.

The compositions and water absorbed for Comparative Examples AO to AW and Examples 18 to 30 are shown in Table 3, the superabsorbent polymer is present in parts by weight based on the weight of the thermoplastic superabsorbent polymer blend composition

and water absorption is reported as grams of distilled water absorbed per gram of superabsorbent polymer.

Table 3

5

Example	Com. Ex.	Thermoplastic Resin	CABLOC 850-13, parts	CABLOC 80HS, parts	CABLOC 88HS, parts	Water Absorption, g
	AO	SURLYN 1702				0
18		SURLYN 1702	35			3
19		SURLYN 1702	45			212
20		PRIMACOR blend (a)	40			214
21		ELVALOY EP4924	40			231
22		PRIMACOR 3460	40			239
23		SURLYN 1702		20		2
24		PRIMACOR blend (a)		20		1
25		PRIMACOR blend (a)		30		2
	AP	PRIMACOR blend (a)				0
26		PRIMACOR blend (b)		40		126
	AQ	ELVALOY EP4924				0
27		ELVALOY EP4924		20		2
28		PRIMACOR 3460		40		118
	AR	XUS60751.17				0
29		SURLYN 1702			35	1
	AS	PRIMACOR 1430				0
30		SURLYN 1702			45	96
	AT	PRIMACOR 5980				0
	AU		100			172
	AV			100		198
	AW				100	153

(a) 50:50 blend of PRIMACOR 3460 / PRIMACOR 5980

(b) 50:50 blend of PRIMACOR 1430 and XUS 60751.17 (EAA with 20.5% PAA)

#### 10 Thermoplastic Superabsorbent Polymer Blended with Polyethylene

In Examples 31 to 38 thermoplastic superabsorbent polymer is melt blended in a Brabender Plasticoder with a 70:30 LLDPE:LDPE polymer blend. The thermoplastic superabsorbent polymer comprises 40 weight percent CABLOC T5066-F which is a sodium polyacrylate superabsorbent polymer that is surface cross-linked having a particle size distribution of about 1 to about 60 micrometers available as a powder from Stockhausen and supplied by the Stewart Superabsorbents LLC and 60 weight percent of a 50:50 PRIMACOR 5980:PRIMACOR 3460 polymer blend. The Brabender Plasticoder conditions are: Barrel temperature is set at 275°F; Mixing RPM is 80; and Mixing times range from 1.5 to 2 minutes. A melt indexer is used to determine the melt flow rate, melt

tension and melt draw down rate of the polymer blends. The blend compositions are considered extrudable. The compositions of Examples 31 to 38 and their MFR, melt tension and draw rates are given in Table 4.

Table 4

Example	Thermoplastic Resin: Superabsorbent Polymer "A"	LLDPE: LDPE polymer blend "B"	Blend Ratio A:B	MFR, Condition	MFR, g/10 min	Melt Tension, units	Draw Rate, fpm	Extrudable
31	60:40 PRIMACOR BLEND: CABLOC T5066F	70:30 LLDPE: LDPE	90:10	C	3.51	0.7	50	Yes
32	60:40 PRIMACOR BLEND: CABLOC T5066F	70:30 LLDPE: LDPE	80:20	C	4.43	0.8	48	Yes
33	60:40 PRIMACOR BLEND: CABLOC T5066F	70:30 LLDPE: LDPE	60:40	C	6.01	0.9	48	Yes
34	60:40 PRIMACOR BLEND: CABLOC T5066F	70:30 LLDPE: LDPE	20:80	C	6.17	1.0	34	Yes
35	60:40 PRIMACOR BLEND: CABLOC T5066F	70:30 LLDPE: LDPE	90:10	E	24.7	0.5	100	Yes
36	60:40 PRIMACOR BLEND: CABLOC T5066F	70:30 LLDPE: LDPE	80:20	E	25.7	0.6	100	Yes
37	60:40 PRIMACOR BLEND: CABLOC T5066F	70:30 LLDPE: LDPE	60:40	E	28.1	0.7	100	Yes
38	60:40 PRIMACOR BLEND: CABLOC T5066F	70:30 LLDPE: LDPE	20:80	E	21.9	0.8	100	Yes

PRIMACOR BLEND: 50:50 blend of PRIMACOR 3460/PRIMACOR 5980

LLDPE = linear low density polyethylene

LDPE = low density polyethylene

Condition C = 150°C/2.16 kg

Condition E = 180°C/2.16 kg

### Monolayer Films

Examples 39 to 42 are monolayer films of thermoplastic superabsorbent polymer blend compositions produced using a cast line process. The thermoplastic superabsorbent polymer blend compositions comprise a thermoplastic resin and CABLOC 850-13. The temperature zones for the cast film process range from 250°F to 320°F. The feedblock and die temperatures range from 270°F to 320°F. Smooth to textured uniformed film having a thickness greater than 6.0 mils or web film having a thickness less than 6.0 mils can be made depending on the take up speed.

The compositions and properties of monolayer films Examples 39 to 42 are shown in Table 5, the superabsorbent polymer is present in parts by weight based on the weight of the thermoplastic superabsorbent polymer blend composition. Absorption capacity in pure water was determined by as described hereinabove.

Table 5

Example	Thermoplastic Resin	CABLOC 850-13, parts	Water Absorption, g
39	SURLYN 1702	35	25
40	SURLYN 1702	45	226
41	PRIMACOR blend (a)	40	219
42	ELVALOY EP4924	40	238

(a) 50:50 blend of PRIMACOR 3460 / PRIMACOR 5980

### Monolayer Films Containing Surfactant

Examples 43 to 46 are mono layer films containing a surfactant. The thermoplastic superabsorbent polymer is melt blended in a Brabender Plasticoder with a commercially available polyethylene containing surfactant compound. The polyethylene containing surfactant is available from AMPACET as ANTIFOG PE MB and contains 10 weight percent active surfactant, mono- and di- glycerides, in a LLDPE/LDPE base polymer. The thermoplastic superabsorbent polymer comprises 40 weight percent CABLOC T5066-F a sodium polyacrylate superabsorbent polymer that is surface cross-linked having a particle size distribution of about 1 to about 60 micrometers available as a powder from Stockhausen and supplied by the Stewart Superabsorbents LLC and 60 weight percent of a 50:50 PRIMACOR 5980:PRIMACOR 3460 polymer blend. The Brabender Plasticoder conditions are: Barrel temperature is set at 275°F; Mixing RPM is 80; and Mixing times

range from 1.5 to 2 minutes. Water absorption and rate of absorption is measured by placing a 2 inch disc sample of a 5 to 7 mil compression molded film in a 2 inch diameter cylinder. At the bottom of the cylinder is a fine mesh screen that is 75 micronmeter or less. A Teflon disk is placed on top of the film sample to secure it in place during the testing.

- 5 The cylinder containing the sample is placed on top of 4 inch glass fret so that the film sample and screen faced the glass fret. A filter paper is placed between the cylinder and the glass fret. The glass fret, filter and the cylinder is placed in a container that contains water so that the height of the water reaches the height of the glass fret. The water continuously being removed and replenished. The entire set up sits on a Mettler PG3001-S balance.
- 10 Once the cylinder containing the sample is placed on the balance, the balance is tared and water absorption and water absorption rate data is generated using a Mettler BalanceLink data acquisition software package. Table 6 lists the compositions for Examples 43 to 46 and their water adsorption amounts and rates.

15

Table 6

Example	Thermoplastic superabsorbent polymer, parts	AMPACET polymer, parts	Water Absorption, g	Time to reach 50% of absorption value, sec	Time to reach maximum absorption value, sec
43	100	0	1	55	225
44	90	10	2.2	40	80
45	80	20	2.6	60	120
46	20	80	1.4	45	80

Thermoplastic superabsorbent polymer = 60 weight percent 50:50 PRIMACOR 5980;PRIMACOR 3460 polymer blend + 40 weight percent CABLOC T5066-F

## 20. Multilayer Films

- Comparative Examples AX to AZ and Examples 47 to 49 are multilayer films of thermoplastic superabsorbent polymer blend compositions produced using a blown film process. The extruder temperature zones for the thermoplastic superabsorbent polymer blend composition (layer 1) range from 250°F to 300°F. Depending on the polymer used,
- 25 the extruder temperature zones for layers 2 and 3 range from 250°F to 400°F and die temperatures range from 250°F to 400°F. The compositions and descriptions of multilayer blown film Comparative Examples AX to AZ and Examples 47 to 49 are shown in Table 7.

Examples 50 to 53 are multilayer blown films prepared as described herein above wherein the level of CABLOC 850-13 is varied in a PRIMACOR blend resin while the composition and ratios of layers 2 and 3 are kept constant. The absorption capacity as described hereinabove and the time to gel block in pure water is determined. The time for the superabsorbent polymer to gel the water at its absorption capacity in pure water for the superabsorbent films, referred to as gel block, is measured according to the following procedure. A sample of the thermoplastic superabsorbent film composition comprising 0.15 gram of superabsorbent polymer in a vial containing 25.6 grams of distilled water. The mixture was shaken by hand until it was gel blocked. The swell initiation time is the time from when the water is added to the first observable swelling of the superabsorbent polymer.

Table 8 lists the compositions and film gauge for multilayer films Comparative Example AAA and Examples 50 to 53. Table 9 lists the water absorption, swell initiation time and time to gel block properties for multilayer films Comparative Examples AAA and Examples 50 to 53 and neat CABLOC 850-13 (Comparative Example AAB).

Table 7

Example	Com. Ex.	Layer 1 composition	Layer 2 composition	Layer 3 composition	Layer ratio			Gauge	Product description
					1	2	3		
	AX	20 parts CABLOC 1181 80 parts LDPE 681	100% ATTANE 4201	100% PRIMACOR 3330	20	60	20	4.0	Frequent pinholes in film, die face build-up
	AY	30 parts CABLOC 1181 70 parts ELVAX 3180	20% ATTANE 4201 80 % LDPE 681	100% PRIMACOR 3330	20	60	20	2.0	Frequent pinholes in film, die face build-up
	AZ	30 parts CABLOC 80HS 70 parts PRIMACOR 3330	100% ATTANE 4201	100% PRIMACOR 3330	20	60	20	4.5	Some pinholes in film, die face build-up
47		40 parts CABLOC 80HS 60 parts PRIMACOR 3460	30% ATTANE 4201 70% LDPE 681	100% PRIMACOR 3330	30	50	20	1.0-2.3	Ran well, no pinholes, no die face build-up.
48		40 parts CABLOC 88HS 60 parts ELVALOY 4924	50% ENGAGE 8100 50% LDPE 681	100% PRIMACOR 3330	30	50	20	2.3	Ran well, no pinholes, no die face build-up.
49		40 parts CABLOC 850-13 60 parts PRIMACOR blend (a)	80% ATTANE 4402 20% LDPE 681	100% PRIMACOR 3330	30	50	20	2.3	Ran well, no pinholes, no die face build-up.

(a) 50:50 blend of PRIMACOR 3460 / PRIMACOR 5980



Table 8

Example	Com. Ex.	Layer 1 composition	Layer 2 composition	Layer 3 composition	Layer ratio			Gauge mils
					1	2	3	
	AAA	100% PRIMACOR blend (a)	80% ATTANE 4201 20% LDPE 681	100% PRIMACOR 3330	30	50	20	4.0
50		10 parts CABLOC 850-13 90 parts PRIMACOR blend (a)	80% ATTANE 4201 20% LDPE 681	100% PRIMACOR 3330	30	50	20	2.0
51		20 parts CABLOC 850-13 80 parts PRIMACOR blend (a)	80% ATTANE 4201 20% LDPE 681	100% PRIMACOR 3330	30	50	20	4.5
52		30 parts CABLOC 850-13 70 parts PRIMACOR blend (a)	80% ATTANE 4201 20% LDPE 681	100% PRIMACOR 3330	30	50	20	1.0-2.3
53		40 parts CABLOC 850-13 60 parts PRIMACOR blend (a)	80% ATTANE 4201 20% LDPE 681	100% PRIMACOR 3330	30	50	20	2.3

(a) 50:50 blend of PRIMACOR 3460 / PRIMACOR 5980

Table 9

Example	Com. Ex.	Water Absorption, g	Swell Initiation Time, sec	Time to Gel Block, sec
	AAA	0		
50		127.25	< 15	Did not gel block
51		203.3	< 15	840-900
52		225.9	< 10	360-420
53		257.65	< 5	90-200
	AAB	180.0	< 5	60-90

Multilayer Films Coated with a Surfactant Solution

Examples 54 to 57 use a 2.0 mil multilayer blown film. The multilayer film comprises as layer 1 a thermoplastic superabsorbent polymer blend comprising 60 weight percent of a 50:50 blend of PRIMACOR 3460 / PRIMACOR 5980 and 40 weight percent CABLOC T5066 F, as layer 2 a LDPE 4005 and as layer 3 PLEXAR 107 an EVA g-MAH from Equistar. The extruder temperature zones for the thermoplastic superabsorbent polymer blend composition (layer 1) range from 250°F to 300°F, the zone temperatures for layer 2 range from 305°F to 310°F and the zone temperatures for layer 3 range from 350°F to 370°F. The thickness ratio for layers 1:2:3 is 30:50:20. Layer 1, the thermoplastic superabsorbent layer, of the multilayer film is sprayed with a surfactant solution ranging from 0 to 8 percent surfactant. The surfactant used for the study is an alcohol ether sulfate. After the film is sprayed, it is placed in an air circulating oven to dry at a temperature of 50°C for 1-2 minutes. Water absorption and rate of absorption is measured according to procedures in the aforementioned section. Table 10 summarizes the water absorption amounts and rates for Examples 54 to 57.

Table 10

Example	Surfactant solution, %	Water Absorption, g	Time to initial absorption, sec	Time to reach 50% of absorption value, sec	Time to reach maximum absorption value, sec
54	0	1.7	15	60	170
55	2	2.0	0	31	112
56	5	1.9	0	29	160
57	8	1.8	0	27	135

20 Superabsorbent Film and Metal Laminate

Example 58 is the multilayer film described in Example 53 laminated to 6.0 mils Electrically Chrome Coated Steel (ECCS) via a heat lamination process. The adhesive layer of the film (layer 3) is used to bond the film to the steel surface. The superabsorbent film/metal laminate can find usefulness in power cable and communication cable construction. The metallic substrate can provide shielding and the thermoplastic superabsorbent polymer layer can be used to bond to itself or another substrate and can

function to stop, block and absorb water in cables. Table 11 shows the adhesion properties for Example 57 superabsorbent film and metal laminate.

Table 11

Example	Film	Metal Type	Peel Strength (a), (lb/in)	Heat Seal (a) Strength, (lb/in)	Jacket (b) Bond Strength, (lb/in)
58	Example 53	ECCS	5.0	13.1	31.8

(a) Peel strength and heat seal strength are measured according to ASTM B 736 and heat seal strength is the bond strength of the thermoplastic superabsorbent polymer to itself.

(b) Jacketing material is DFDD 6069 BK 9865 a modified LLDPE which is a standard wire and cable jacketing resin manufactured by Union Carbide. Jacket bond (composite of jacketing material and laminate fabricated in a platen press) strength measures the force to separate the jacket from the laminate, values is measured according to ASTM D 4365-86 modified to hold the sample at 180°C.

ECCS = electrically chrome coated steel

#### Armored Cable

Superabsorbent films were laminated to the ECCS and slit into 2.25 inch wide steel tape. The tape is used to make armored cables Examples 59 to 62. The steel tape is corrugated to 32 corrugations per inch (corrugation can be achieved with or without oil). The corrugated tape is longitudinal formed through a series of forming dies. A PVC jacketed insulated copper pair cable core having an outside diameter of 0.60 inch is placed inside the formed armored tape. A jacketing resin is then extruded onto the formed armor tape to make a final cable having a final outside diameter of 0.742 inch. The final gap between the inner jacket and the armor tape is calculated to be around 0.015 inch (0.381mm).

The performance of cables comprising the thermoplastic superabsorbent polymer laminate (Examples 59 to 62, Table 12) is compared to cables comprising ZETABON CJBS262 armor tape available from the Dow Chemical Company (Comparative Example AAC) and additionally comprising a non-woven superabsorbent tape 3E252 produced by Lantor Inc. (Comparative Example AAD). Non-woven superabsorbent tapes are the wire and cable industry standard for use in dry cable designs. The non-woven superabsorbent tape comprises superabsorbent particles sandwiched between two non-woven materials. For this evaluation, the non-woven superabsorbent tape is helically wrapped around the copper pair cable core before placing the cable core inside the formed armor tape. In the wire and cable industry, the non-woven superabsorbent tape is typically longitudinal formed around the cable core.

Water blocking performance of the cables is determined by the EIA/TIA-455-82A ("L-test"). The end of the cable core is taped or sealed so that water can not migrate through the wires of the cable core. The cable length is 1 meter, test duration is 24 hours, the water column is 1 meter and time to penetration is measured.

5

Table 12

Example	Com. Ex.	Laminate composition			Non-woven tape	Time to penetration
		Film Layer 1	Metallic Core	Film Layer 2		
	AAC	EAA film	6 mil ECCS	EAA film	No	Within 1 minute
	AAD	EAA film	6 mil ECCS	EAA film	Yes	(a)
59		EAA film	6 mil ECCS	Film 1	No	No penetration
60		EAA film	6 mil ECCS	Film 2	No	No penetration
61		EAA film	6 mil ECCS	Film 3	No	No penetration
62		EAA film	6 mil ECCS	Film 4	No	No penetration

(a) test results vary from no penetration to penetration occurring between 15-24 hours

EAA film = 90% PRIMACOR 3330 / 10% PE

10

Film 1 composition: Layer 1: 30% - 40 parts CABLOC 850-13 / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)  
 Layer 2: 50% - 80% ATTANE 4201 / 20% LDPE 681  
 Layer 3: 20% - PRIMACOR 3330

15

Film 2 composition: Layer 1: 30% - 40 parts CABLOC 80HS / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)  
 Layer 2: 50% - 80% ATTANE 4201 / 20% LDPE 681  
 Layer 3: 20% - PRIMACOR 3330

20

Film 3 composition: Layer 1: 30% - 40 parts CABLOC 1181 / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)  
 Layer 2: 50% - 80% ATTANE 4201 / 20% LDPE 681  
 Layer 3 20% - PRIMACOR 3330

Film 4 composition: Layer 1 30% - 40 parts CABLOC 80HSB / 60% (50/50 PRIMACOR 3460/PRIMACOR 5980)  
 Layer 2 50% - 80% ATTANE 4201 / 20% LDPE 681  
 Layer 3 20% - PRIMACOR 3330

CABLOC 80HSB has a particle size distribution from about 1 to about 20 micrometers

Film Layer 1 is the side of the laminate that is used to bond to the jacketing resin

Film Layer 2 is the side of the laminate that was facing the core

Armor Cable with Thermoplastic Superabsorbent Polymer Coated with a Surfactant

Superabsorbent films were laminated to the ECCS. The superabsorbent layer of the film is either pre- or post-coated with an alcohol ether sulfate surfactant solution. The concentration of the surfactant solution ranges from 2 weight percent to 8 weight percent.

5 An antifoaming agent, Dow Corning Anti Foam 1520-US, is also used. The amount of antifoam used is 2500 ppm. The coated laminate is slit into 1.375 inch wide steel tape. The tape is used to make armored cables Examples 63 to 68 (Table 13). The steel tape is corrugated to 32 corrugations per inch (corrugation can be achieved with or without oil). The corrugated tape is longitudinal formed through a series of forming dies. An HDPE core tube, available from United States Plastic Corporation, having an outside diameter of 0.375  
10 inch is placed inside the formed armored tape. A jacketing resin is then extruded onto the formed armor tape to make a final cable. The final gap between the inner jacket and the armor tape is calculated to be around 0.020 inch (0.508mm).

The performance of cables comprising the thermoplastic superabsorbent polymer laminate (Examples 63 to 68) is compared to cables comprising ZETABON CJBS262  
15 armor tape available from the Dow Chemical Company (Comparative Example AAC).

Water blocking performance of the cables is determined by the EIA/TIA-455-82A ("L-test"). The end of the cable core is taped or sealed so that water can not migrate through the wires of the cable core. The cable length is 1 meter, test duration is 24 hours,  
20 the water column is 1 meter and time to penetration is measured.

Table 13

Example	Com. Ex.	Laminate composition			Surfactant treatment	Time to penetration
		Film Layer 1	Metallic Core	Film Layer 2		
	AAC	EAA film	6 mil ECCS	EAA film		Within 1 minute
63		EAA film	6 mil ECCS	Film 1	Pre-	Pass
64		EAA film	6 mil ECCS	Film 2	Pre-	Pass
65		EAA film	6 mil ECCS	Film 2	Post-	Pass
66		EAA film	6 mil ECCS	Film 2	Post-	Pass
67		EAA film	6 mil ECCS	Film 3	Post-	Pass
68		EAA film	6 mil ECCS	Film 3	Post-	Pass

EAA film = 90% PRIMACOR 3330 / 10% PE

25 Film 1 composition: Layer 1: 30% - 40 parts CABLOC 850-13 / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)  
Layer 2: 50% - LDPE 4005  
Layer 3: 20% - PLEXAR 107

Film 2 composition: Layer 1: 30% - 40 parts CABLOC T5066 F / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)  
Layer 2: 50% - LDPE 4005  
30 Layer 3: 20% - PLEXAR 107

Film 3 composition: Layer 1: 30% - 40 parts Norsocryl XFS / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)  
 Layer 2: 50% - LDPE 4005  
 Layer 3 20% - PLEXAR 107

Norsocryl is a crosslinked copolymers of acrylic acid and sodium acrylate supplied by elf atchem ATO  
 PLEXAR 107 is a grafted maleic anhydride EVA copolymer supplied by Equistar  
 Film Layer 1 is the side of the laminate that is used to bond to the jacketing resin  
 Film Layer 2 is the side of the laminate that was facing the core

### Foam Thermoplastic Superabsorbent Polymer

Examples 69 to 77 are extruded foams of thermoplastic superabsorbent polymer blend compositions. About 12 parts per hundred (pph) HCFC 142B physical blowing agent is used. The extruder temperature zones range from 110°C to 150°C and the die temperature range from 85°C to 90°C. The compositions and description of the foam are shown in Table 14. The resulting foams are soft, flexible and non-friable. The superabsorbent particulates are uniformly distributed on the skin and throughout the cell structure of the foam.

Table 14

Example	Thermoplastic superabsorbent polymer type	Foam type
69	1	Semi-porous to closed cell foam
70	2	Semi-porous to closed cell foam
71	3	Semi-porous to closed cell foam
72	4	Semi-porous to closed cell foam
73	5	Semi-porous to closed cell foam
74	6	Semi-porous to closed cell foam
75	7	Semi-porous to closed cell foam
76	8	Semi-porous to closed cell foam
77	9	Semi-porous to closed cell foam

1 composition: 10 parts CABLOC T5066 F / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

2 composition: 20 parts CABLOC T5066 F / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

3 composition: 30 parts CABLOC T5066 F / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

4 composition: 30 parts CABLOC 80 HS / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

5 composition: 20 parts CABLOC HCF / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

6 composition: 20 parts Norsocryl XFS / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

7 composition: 20 parts Norsocryl S35 / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

8 composition: 30 parts Norsocryl S35 / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

9 composition: 35 parts Norsocryl S35 / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

Norsocryl is a crosslinked copolymers of acrylic acid and sodium acrylate supplied by elf atchem ATO

Norsocryl XFS particle size distribution range from 1-67 micronmeter

Norsocryl S25 particle size distribution range form 1-225 micronmeter

The absorption capacity in pure water (WAC) of thermoplastic superabsorbent foam Examples 78 to 80 (Table 15) extruded by the abovementioned extrusion foam process is shown in Table 16. The WAC is measured according to the following procedure: the foam is cut in 0.125 inch by 0.625 in by 0.125 to 0.25 inch and an amount of the cut foam sample determined to contained 0.1 gram of the superabsorbent polymer (based on the percent superabsorbent polymer in the foam composition) weighing W1 is placed in 0.150 liter of distilled water and is shaken on a shaker for 2 hours. The water is filtered from the foam through a 75 micrometer sieve. The weight of the swollen foam (W2) is then measured. The amount of water absorbed, (Wa) is calculate by the following formula:

$$W_a = (W_2 - W_1) * 10$$

Table 15

Example	Thermoplastic superabsorbent polymer type	Foam	Water absorption, g
78	2	Yes	87
79	3	Yes	67
80	3	Yes	43

2 composition: 20 parts CABLOC T5066 F / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

3 composition: 30 parts CABLOC T5066 F / 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

9 composition: 35 parts Norsocryl S35/ 60 parts (50/50 PRIMACOR 3460/PRIMACOR 5980)

From these data it can be concluded that the extrudable thermoplastic superabsorbent polymer blends of the present invention comprising one or more superabsorbent polymer and one or more thermoplastic resin wherein the thermoplastic resin comprises a functional group that interacts with the superabsorbent polymer yields the best balance of superabsorbent polymer containment, processability, formability and absorption properties.

It has been found that the present invention provides improved thermoplastic superabsorbent polymer blend compositions and processes for preparing, among other things, monolayer films, multilayer films, nonwoven webs, sheets, foams, profiles, multilayer laminates, fibers, tubes, rods, and pipes. It can be seen that the resulting parts or structures according to the present invention are surprisingly improved by the use of the

described extrudable thermoplastic superabsorbent polymer blend compositions and that extruded, shaped or otherwise fabricated articles will ease manufacture, improve performance and reduce costs of absorbent articles constructed therefrom.



## CLAIMS:

1. An extrudable thermoplastic superabsorbent polymer blend composition comprising
  - (a) one or more superabsorbent polymer and
  - 5 (b) one or more thermoplastic resin comprising a functional group which interacts ionically or covalently with (a).
2. The extrudable thermoplastic superabsorbent polymer blend composition of Claim 1 having a melt draw down rate between about 5 and about 100 feet per minute and a melt tension between about 0.1 and about 10 under temperature and applied load conditions  
10 that give a melt flow rate of between about 0.1 and about 300 g/10 min.
3. The extrudable thermoplastic superabsorbent polymer blend composition of Claim 1 wherein the superabsorbent polymer is prepared from water-soluble  $\alpha,\beta$ -ethylenically unsaturated monomers.
4. The extrudable thermoplastic superabsorbent polymer of Claim 3 wherein the  
15  $\alpha,\beta$ -ethylenically unsaturated monomers is a monocarboxylic acid, a vinyl polycarboxylic acid, an acrylamide or mixtures thereof.
5. The extrudable thermoplastic superabsorbent polymer blend composition of Claim 1 wherein the superabsorbent polymer is a cellulosic-graft copolymer, a starch-graft copolymer, a starch-g-poly(acrylic acid), a polyacrylamide; a polyvinyl alcohol, a  
20 poly(acrylic acid), a copolymer of sulfonic acid group containing monomer, or mixtures thereof.
6. The superabsorbent polymer of Claim 5 is crosslinked, partially neutralized, surface treated or combinations thereof.
7. The extrudable thermoplastic superabsorbent polymer blend composition of  
25 Claim 1 wherein the thermoplastic resin is a polyacrylic acid, ethylene and acrylic acid copolymer, ethylene, t-butylacrylate and acrylic acid terpolymer, ethylene and methacrylic acid copolymer, ionomers of ethylene and methacrylic acid copolymers, ethylene, vinyl acetate and carbon monoxide terpolymer, ethylene and carbon monoxide copolymer, ethylene, acrylic acid and carbon monoxide terpolymers, ethylene, n-butylacrylate and  
30 carbon monoxide terpolymer or blends thereof.
8. The extrudable thermoplastic superabsorbent polymer blend composition of Claim 1 further comprising a surfactant.

9. The extrudable thermoplastic superabsorbent polymer blend composition of Claims 1, 3 or 8 further comprising a polyethylene, a copolymer of polyethylene, a polypropylene, a copolymer of polypropylene or a polystyrene.

10. A method for preparing an extrudable thermoplastic superabsorbent polymer blend composition comprising the step of combining:

- (a) one or more superabsorbent polymer and
- (b) one or more thermoplastic resin comprising a functional group which interacts ionically or covalently with (a).

11. The method of Claim 10 further comprising the step of combining (c) a surfactant.

12. A method for producing an extruded or molded article of an extrudable thermoplastic superabsorbent polymer blend composition comprising the steps of:

1) preparing an extrudable thermoplastic superabsorbent polymer composition comprising

- (c) one or more superabsorbent polymer and
- (b) one or more thermoplastic resin comprising a functional group which interacts ionically or covalently with (a) and

2) extruding or molding said thermoplastic superabsorbent polymer composition into an extruded or molded article.

13. The method of Claim 12 wherein the superabsorbent polymer composition further comprising (c) a surfactant.

14. The method of Claims 12 or 13 wherein the extruded article is a monolayer film, a multilayer film, a nonwoven web, a sheet, a foam, a profile, a multilayer laminate, a fiber, a tube, a rod or a pipe.

15. The method of Claims 12 or 13 wherein the extruded article is a monofilament fiber, a bicomponent monofilament fiber, a spun bond nonwoven web, a melt blown nonwoven web, or a composite comprising combinations thereof.

16. The method of Claims 12 or 13 wherein the extruded article is a nonwoven web comprising a spun bond nonwoven web comprising one or more bicomponent fiber, a melt blown nonwoven web comprising one or more bicomponent fiber, or a composite structure comprising at least one layer of one or more spun bond nonwoven web and at least one layer of one or more melt blown nonwoven web wherein one or more layers of the composite comprise bicomponent fibers.

17. The composition of Claims 1 or 8 in the form of an extruded or molded article.

18. The extruded or molded article of Claim 17 is a monolayer film, a multilayer film, a nonwoven web, a sheet, a foam, a profile, a multilayer laminate, a fiber, a tube, a rod or a pipe.

5 19. The extruded or molded article of Claim 17 is a monofilament fiber, a bicomponent monofilament fiber, a spun bond nonwoven web, melt blown nonwoven web, or a composite comprising combinations thereof.

20. The extruded or molded article of Claim 17 is a nonwoven web comprising a spun bond nonwoven web comprising one or more bicomponent fiber, a melt blown  
10 nonwoven web comprising one or more bicomponent fiber, or a composite structure comprising at least one layer of one or more spun bond nonwoven web and at least one layer of one or more melt blown nonwoven web wherein one or more layers of the composite comprise bicomponent fibers.

21. The monolayer film or multilayer film of Claim 18 laminated to a metal.

15 22. A power cable comprising the metal laminate of Claim 21.

23. A communications cable comprising the metal laminate of Claim 21.

24. A power cable comprising the monolayer film or multilayer film of Claim 18.

25. A communications cable comprising the monolayer film or multilayer film of Claim 18.

20 26. A disposable absorbent device comprising an extruded or molded article of Claim 18.

27. The disposable absorbent device of Claim 26 is a diaper, a sanitary napkin, a tampon, an incontinence product, a hospital gown or a bed pad.

25 28. A disposable absorbent device comprising an extruded or molded article of Claim 19.

29. The disposable absorbent device of Claim 28 is a diaper, a sanitary napkin, a tampon, an incontinence product, a hospital gown or a bed pad.

30. A disposable absorbent device comprising an extruded or molded article of Claim 20.

30 31. The disposable absorbent device of Claim 30 is a diaper, a sanitary napkin, a tampon, an incontinence product, a hospital gown or a bed pad.

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- (74) Agent: CHRISTY, M., Robert; The Dow Chemical Company, Intellectual Property, P.O. Box 1967, Midland, MI 48641-1967 (US).
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(54) Title: THERMOPLASTIC SUPERABSORBENT POLYMER BLEND COMPOSITIONS AND THEIR PREPARATION

(57) Abstract: An extrudable thermoplastic superabsorbent polymer blend composition is disclosed. The blend compositions are especially well suited for preparation of extruded or molded articles such as monolayer films, multilayer films, nonwoven webs, sheets, foams, profiles, multilayer laminates, fibers, tubes, rods or pipes which in turn are well suited for preparation of power and communication cables or disposable absorbent articles such as diapers, sanitary napkins, tampons, incontinence products, hospital gowns or bed pads.

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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, COMPENDEX

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X	WO 99 57201 A (FULLER H B LICENSING FINANC ;AHMED SHARF U (US); CLAPP LESLIE J (U) 11 November 1999 (1999-11-11) claims 1,2,10,13,20,24 ---	1,7,10, 14,26
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A	EP 0 272 682 A (KIMBERLY CLARK CO) 29 June 1988 (1988-06-29) claim 1 ---	1
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☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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A	<p>EP 0 338 393 A (KIMBERLY CLARK CO)  25 October 1989 (1989-10-25)  claim 1</p> <p>-----</p>	1

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